MOLYBDENUM (ATOMIC ABSORPTION, DIRECT ASPIRATION)

1.0 SCOPE AND APPLICATION

1.1 See Section 1.0 of Method 7000.

2.0 SUMMARY OF METHOD

2.1 See Section 2.0 of Method 7000.

3.0 INTERFERENCES

3.1 See Section 3.0 of Method 7000 if interferences are suspected.

3.2 Interferences in an air/acetylene flame from Ca, Sr, SO_4, and Fe are severe. These interferences are greatly reduced in the nitrous oxide flame and by addition of 1,000 mg/L aluminum to samples and standards.

4.0 APPARATUS AND MATERIALS

4.1 For basic apparatus, see Section 4.0 of Method 7000.

4.2 Instrument parameters (general):

4.2.1 **Molybdenum hollow cathode lamp.**
4.2.2 **Wavelength:** 313.3 nm.
4.2.3 **Fuel:** Acetylene.
4.2.4 **Oxidant:** Nitrous oxide.
4.2.5 **Type of flame:** Fuel rich.
4.2.6 **Background correction:** Required.

5.0 REAGENTS

5.1 See Section 5.0 of Method 7000.

5.2 Preparation of standards:

5.2.1 **Stock solution:** Dissolve 1.840 g of ammonium molybdate, $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ (analytical reagent grade), in Type II water and dilute to 1 liter; 1 mL = 1 mg Mo (1,000 mg/L). Alternatively, procure a certified standard from a supplier and verify by comparison with a second standard.

5.2.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed after processing. The samples and standards should also contain 1,000 mg/L aluminum (see Paragraph 5.2.3).

5.2.3 **Aluminum nitrate solution:** Dissolve 139 g aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, in 150 mL of Type II water; heat to effect solution. Allow to cool and make up to 200 mL. To each 100 mL of standard and sample alike, add 2 mL of the aluminum nitrate solution.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See Chapter Three, Section 3.1.3, Sample Handling and Preservation.

7.0 PROCEDURE

7.1 Sample preparation: The procedures for preparation of the sample are given in Chapter Three, Section 3.2.

7.2 See Method 7000, Paragraph 7.2, Direct Aspiration.

8.0 QUALITY CONTROL

8.1 See Section 8.0 of Method 7000.

9.0 METHOD PERFORMANCE

9.1 The performance characteristics for an aqueous sample free of interferences are:

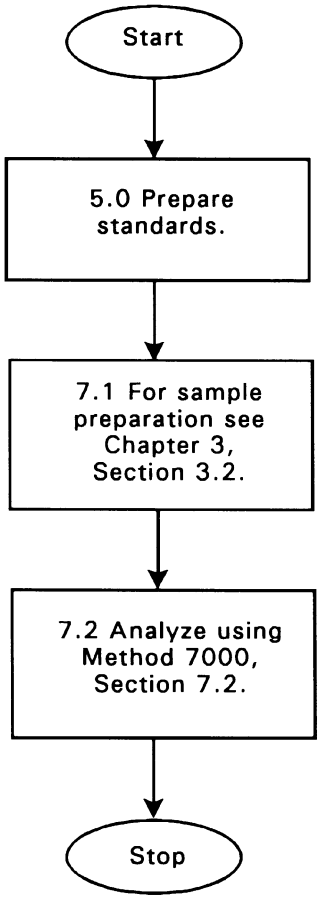
Optimum concentration range: 1-40 mg/L with a wavelength of 313.3 nm.
Sensitivity: 0.4 mg/L.
Detection limit: 0.1 mg/L.

9.2 In a single laboratory, analysis of a mixed industrial-domestic waste effluent, digested with Method 3010, at concentrations of 0.3, 1.5, and 7.5 mg/L gave standard deviations of $\pm$0.007, $\pm$0.02, and $\pm$0.07, respectively. Recoveries at these levels were 100%, 96%, and 95%, respectively.

9.3 For concentrations of molybdenum below 0.2 mg/L, the furnace technique (Method 7481) is recommended.

10.0 REFERENCES

1. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December 1982, Method 246.1.


Start
5.0 Prepare standards.
7.1 For sample preparation see Chapter 3, Section 3.2.
7.2 Analyze using Method 7000, Section 7.2.
Stop